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# Separation of maize particles from alcohol extracts with minimal losses☆

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**Abstract**

Low-cost extraction of maize protein using ethanol at the front end of a dry grind ethanol plant has been discussed in recent articles. Thorough recovery of (alcoholic) extract from the extracted maize is essential to make the process economical and to prevent the ethanol from interfering with liquefaction enzymes or fermentation organisms where the solid residues are to be subsequently reduced to glucose and finally fermented to ethanol. Because the particles will be converted in water it is unnecessary to dry them, if a (miscible) liquid/liquid miscible displacement method can be used. Therefore, three methods of displacing extract liquid from the extracted corn particles were tried using (1) packed bed displacement, (2) centrifugation with rinsing, and (3) gravitational settling into water. Displacing extract (liquid) from a stationary bed of milled corn extract was too slow to be practical. This finding should discourage the use of similar techniques based on stationary layers of extracted corn. Continuous centrifugation, with rinsing by fresh ethanol solution, was effective at recovering protein in the extract with rinse rates comparable to extract feed rates. Settling the extracted corn into water appears to be the most feasible method of separation, requiring low extract dilution and inexpensive equipment. 

*Keywords:* Maize; Extraction; Ethanol; Solvent recovery; Centrifugation; Gravitational particle settling

## 1. Introduction

Prolamine-rich, water insoluble proteins (zein) can be extracted from milled maize by vigorous mixing in heated ethanol solutions. Although a relatively small amount of zein is produced from corn gluten, no other zein type proteins are commercially extracted from grain. Recent studies suggest that extraction of zein from maize could be commercially feasible, as part of an ethanol plant (Dickey et al., 1999; Shukla et al., 2000).

To minimize cost, as little ethanol as possible should be lost during separation of the maize solids from the extract mixture. Although the extract liquid remaining on the solid particles (after liquid/solid separation) can be recovered by evaporation and condensation, this process

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would be expensive. No examples of thorough separation of solvent from agriculturally derived particles, other than by evaporation, are known to the authors. The extract solids can be separated from the solvent in several ways. We investigated three methods including (1) rinsing a packed bed formed by collection of the solids in a column, (2) rinsing during continuous centrifugation and (3) settling from the extract mixture into water.

Experience with ethanolic maize extracts has shown that most of the solvent can be separated from the extract mixture using either a decanter centrifuge, which produces a solids-rich product containing about 55% solids, or a screen, which produces a 20–40% solids product (depending on the screen, the feed and feed rate). A recent design study proposed that a continuous cake filter would be the most appropriate device to separate alcohol from 40 μm potato starch granules (Tijsen et al., 2001). Potato starch granules are similar in size to the particles in an extracted, milled corn mixture. In all these cases, the liquid clinging to the surface of the particles is being recovered and not the liquid absorbed into the particles. The absorbed liquid can only be recovered by evaporation, which will usually not be attractive because of the small amount absorbed by starch granules.

Before the extracted maize in the solids-enriched stream from a decanter centrifuge, filter or screen is pumped to the starch liquefaction vessel, the residual extract (liquid) in the stream should be removed from the solids, to keep it from interfering with liquefaction and saccharification enzymes or with yeast in the fermentor. Thorough removal of the extract liquid will enable recovery of most of the ethanol solution and zein solute.

The feasibility of the first two methods tried depends on the susceptibility of the collected solids to rinsing. When the extract liquid is displaced from extracted maize solids with water, evaporation of the displacing water is unnecessary because the starch solids will be subsequently processed (liquefaction, saccharification, and finally fermentation) in aqueous slurry. However, water may dilute the extract liquid at the water/extract interface enough to precipitate dissolved zein, thus rendering the particle layer less permeable and more likely to trap extract liquid. This solute precipitation can be avoided by displacing the liquid twice, first with a liquid similar to the original and free of precipitatable solute, and second with water. Double displacement is needed when using a method where the particles have been concentrated and are close together, and not when they are dispersed, such as when the particles settle from the original extract.

## 2. Materials and methods

### 2.1. Maize preparation

The maize used was a yellow dent maize meal produced by a commercial feed mill (Davis Feed Mill, Perkasie, PA). The shelled maize was cracked with a roller mill and the pericarp removed by aspiration. The cracked maize was passed through a counter-rotating, ribbed disc mill and reduced to a median size of 0.35 mm. The median size of the meal particles was 0.35 mm based on sieve separation of 23 kg. Although it was not possible to control the maize hybrid purchased from the mill, all milled corn used for a series of extractions was purchased at the same time and stored in a cold chamber at −20 °C until it was extracted.

### 2.2. Analysis

The protein content (N × 6.5) was determined by the micro-Kjeldahl method (AACC, 1995; AOAC, 1995). Lipid content was determined by blending samples of between 100 and 300 mg with a total of 125 ml of chloroform:methanol (2:1, v/v) for approximately 2 min. The liquid extract was filtered through coarse sintered glass funnels and dried under nitrogen at room temperature. The dried extracts were re-dissolved in 5–10 ml of chloroform and transferred to tared vials. Samples were filtered through glass wool (previously washed with 2:1 chloroform:methanol) if they contained insoluble material such as starch. The chloroform solution was dried to a constant weight with nitrogen and the lipid weight determined. Solid content was determined by weighing before and after heating to 100 °C overnight. Ethanol content of extract solutions and diluted

centrifugates was calculated from liquid density and temperature measurements. Solution densities were measured with calibrated hydrometers (Ever Ready Thermometer Co., Inc., West Patterson, NJ). The ethanol concentration calculations were confirmed by HPLC measurements of some samples. The concentrations calculated from the densities agreed with HPLC measurements within 1.5%.

### 2.3. Extraction and extract separation by packed bed rinsing

Maize, 46.2 kg, was extracted for 90 min at 50 °C in the following manner. The milled maize was added to 211 kg of 70% ethanol solution, in a jacketed, pressure tight, 1300-l tank (Paul Mueller Company, Springfield, MO). The slurry was agitated by two scraper blades attached to a central shaft rotating at 200 rpm and circulated out of the tank through a centrifugal pump (Fristram, model FP702, Middletown, WI) and back to the tank, to increase the disruption of the endosperm structure. The temperature of the suspension was raised to 50 °C in a few minutes. It was held at this temperature for 90 min, and then cooled to ambient temperature. This process was used for all extractions described hereafter unless otherwise noted. After cooling, the extract mixture was pumped with a Masterflex L/S pump (Cole-Parmer Instrument Co., Vernon Hills, IL) at 130 kg/h into a vertical glass pipe, 225 mm diameter by 1500 mm long (Q.V.F., Inc., Horseheads, NY). The fluid was admitted to the column through a sealing plate at the top of the column fitted with a hose connection and valve and a separate connection to an air supply. The bottom of the column was fitted with a #14 screen (1.4 mm opening size) above a sealing plate fitted with a valve, which could be opened to allow liquid to drain from the column, through the bed formed by the solid particles collected above the filter. Sufficient slurry was pumped to the column to create a loosely packed bed 118 cm high with 2.5 cm of liquid at the top. The column was allowed to settle overnight and most of the clear liquid (33 cm) was pumped from the top of the column, leaving 85 cm of packed solid particles and extract liquid. A 70% ethanol solution containing methyl blue indicator and 5% 20 K polyethylene glycol (PEG) was prepared to use as a displacing liquid. The PEG was added to insure that the displacing viscosity exceeded that of the extract liquid to reduce fingering. The viscosity of the extract liquid was measured equal to 2.3 cP and the displacing solution was 3.1 cP. A 11-cm layer of the displacing liquid was pumped to the top of the bed and 138 kPa of air pressure applied through the top of the column. The bottom valve was left open and drained liquid collected and weighed periodically. The descent of the blue level through the bed was followed visually.

### 2.4. Rinsing during centrifugation

Three runs were made. For the first run, 46 kg of cold, milled corn, removed from cold storage an hour before extraction, was extracted with 182 kg of 70% ethanol. After cooling to ambient temperature the extract mixture (20% solids on average) was pumped to a decanter centrifuge (Sharples, Division of Alfa Laval, Model P660, Warminster, PA) rotated at 4900 rpm with a backdrive rotating at 2940 rpm. Neither bowl nor backdrive speeds were varied. The extract mixture was fed to the decanter centrifuge at a constant volumetric rate, 70.5 kg/h during the first run, using a Masterflex I/P tubing pump (Cole-Parmer Instrument Co.) and 70% ethanol rinse solution was fed using a Masterflex variable speed L/S tubing pump with 6419-24 tubing at five different rates. The tubing pump readout at 2.8 ml/rpm, specific gravity of 0.87 was used to calculate rinse rates. The liquid product rates were measured by timing a sample of the liquid stream that was subsequently weighed.

The centrifuge generated $3100 \times g$. The centrifuge feed tube was equipped with an annulus through which a rinsing stream was directed into the bowl. The rinse entered the bowl 5.08 cm from the end of the feed tube, upstream from the liquid flow and downstream from the beginning of the solid layer being pushed in the opposite direction. Thus, the rinse stream encounters a damp layer still in the process of consolidation and susceptible to liquid displacement.

Samples of the liquid and solid product streams were collected in plastic containers that were immediately capped and saved for analysis. Rates were calculated from the weight and collection time of samples taken concurrently with samples for compositional analysis. A 5–10 kg sample was typically collected for rate determinations.

A second decanter run was made to gauge the variation of solids content in the extraction tank outflow shown in the first run. For this run, 46.8 kg of milled corn was taken from cold storage on the afternoon preceding its extraction extracted with 182 kg of 70% ethanol as described for the preceding run and the extract fed to the decanter centrifuge at 72.3 kg/h with a 35 kg/h rinse of 70% ethanol. Flow rates from the centrifuge were measured by weighing samples of product collected for a measured time. Liquid and solid samples were taken at the beginning and end of each interval.

For the third run, 46 kg of milled corn was taken from cold storage on the day preceding the extraction, which was the same as the second run. Recovered extract liquid from previous extractions (182 kg) containing 59% ethanol, 0.16% protein, 1.7% solids, and 0.075% lipid by weight was used. After cooling the extract mixture to ambient, it was pumped to the centrifuge at a constant volumetric feed rate of 175 rpm, through #73 tubing (2.2 l/min), equal to 116.8 kg/h. The extracted mixture was pumped to the decanter centrifuge for 1.5 h, and the decanter was also fed a rinse stream of 70% ethanol at 35 kg/h for 27 min in the middle of that period. Three samples of the liquid and solid product streams were taken for the first and last unrinsed and for the rinsed periods.

### 2.5. Settling from the extract into water

The separation was achieved by pumping the extract mixture, from various amounts of milled maize as described subsequently, to a 1200-l, stainless steel, jacketed, slightly inclined from the horizontal, tank (Mojonnier Bros. Co., Chicago, IL) with a jackscrew attached to one end that allowed the tank to be tipped up to 7° d from horizontal. The tank was partly filled with water and a flow distributor comprised of a cage containing a close packed layer of 0.3 cm diameter, floating, polypropylene balls (Small Parts Inc., Miami Lakes, FL) was attached to the lower of two 0.5 m diameter horizontal (when the tank axis is horizontal) circular ports on the top of the tank. The centers of the near-horizontal ports are 76 cm apart, in line with the tank axis. The buoyant balls formed layers that slowed and dispersed the incoming extract mixture that was pumped into the tank above the distributor at 50 kg/h. The function of the balls was to reduce mixing of the water and extract liquid (at the beginning of the extract introduction) while allowing the solids in the extract stream to settle to the water layer. A slightly different floating barrier method was described for the mineral processing industry (Spear, 1980). As the extract mixture was pumped into the tank, a layer of extract liquid formed on top of the water and the solid particles settled through the liquid layers balls to the bottom of the tank. The upper level of the extract liquid was controlled by pumping liquid out of the tank through a tube fixed to the upper port and extending several centimeters into the tank. After all of the extract had been pumped to the tank, and later after the contents had been allowed to settle overnight, the upper layer of liquid was pumped out, in batches, to a small container. The container was weighed and a sample taken. The filling, sampling and weighing process was repeated until all the upper layer of liquid had been removed from the tank. Consequently, the lower, aqueous layer was pumped out, along with the extracted corn, which was screened from the liquid. Each phase was weighed and sampled. The distributor design and mass of extract was changed in the experiments made to test this method.

The distributor used on the initial trial was comprised of a pair of 0.3 m diameter circular plates with 56 holes uniformly distributed on each. The holes in each plate are aligned with those of the other plate and together they loosely confined 56, 1.7 cm diameter plastic balls with densities slightly less than that of ethanol. Two bags of milled corn, 46.8 kg total, were extracted with 182 kg of 70% ethanol. Water (219 kg) was pumped into the tank prior to introduction of the extract

mixture. After all of the extract mixture had been pumped into the settling tank, the upper end was jacked up 40 cm to position the tank at a 6° tilt from the horizontal.

The distributor was modified for later settling tests to allow unhindered vertical travel of the buoyant balls, which were contained within a 29 cm long, 15 cm diameter bottomless cylinder made of an open-mesh copper screen. The settling tank was partly filled with 854 kg of water before pumping extract into it. After extraction of 68 kg of milled corn and cooling, the extract mixture was pumped at 68 kg/h through the distributor mounted on the lower, of two, top ports of the settling tank. The initial water level was about 20 cm below the top of the distributor so the incoming extract dropped that distance before encountering the buoyant balls or the retaining screen. When the extract layer rose to the outlet nozzle, (extract) liquid removal through the upper port was begun. About 168 kg of liquid was removed while the extraction tank was pumped out, after which the settling tank lids were fixed over the ports and the solids were allowed to continue settling for about 18 h. Removal of the upper (extract) liquid layer was completed in batches that were weighed and sampled.

A third test was made with 90.9 kg of maize and 363.6 kg of extraction liquid. A solids laden water stream was continuously removed from the bottom of the settling tank and replaced with an equal volume of water at 1256 kg/h. The settling tank was initially filled with 917 kg of water, at a depth of 68 cm at the lower port. Settled extract (164 kg) was pumped at 132 kg/h, from the upper port, when the extract level was 13 cm above the water level. As before, the rest of the extract (291 kg) was allowed to settle overnight and removed, weighed and sampled in batches the next morning.

## 3. Results

### 3.1. Packed bed rinsing

Pressing of the bed for the first 6 h caused a 15 cm reduction in bed length, a penetration of 500 ml of displacing liquid and drainage of 21 kg of extract liquid. Calculation of the bed permeability gave a value of 10 md (millidarcies), below that of sand (20–200 d) and in the midrange of limestone (0.2–45 md). The bed volume continued to drop with time and the values were fit to an exponential expression the volume fraction compressed = $0.2(1-\exp(-0.14t))$ with $t$ in h.

The displacement rate dropped to a rate of 0.5 kg/h after about 80 h, which was maintained until 140 h, when the test was stopped. After the displacement stopped a sample of the damp displaced corn was immersed in water and its specific gravity determined to be 1.09; taking the starch and liquid specific gravities to be 1.3 and 0.87 and additivity of the mass of these components, the solids content of the displaced bed was calculated to be 50%.

### 3.2. Rinsing during centrifugation

The average liquid recovery rate for the first run, based on measured samples, was 71 kg/h above the non-rinsed sample output, 58 kg/h. The tubing pump readout at 2.8 ml/rpm, specific gravity of 0.87 was used to calculate rinse rates. The product liquid rates were: 81, 58, 104, 95 and 110 kg/h. The extract mass feed rates calculated from the measured output varied slightly due to an increase in solids content in the feed from the tank at the end of the transfer period.

The liquid product stream (decantate) concentration depends on the fraction of rinse flow that displaces liquid extract from solids prior to the expression of the solid product stream. This fraction, $X$, can be calculated from a mass balance:

$$X = ((\mathrm{RSC}/\mathrm{USC})(\mathrm{RFR}) - \mathrm{UFR})/(\mathrm{RFR} - \mathrm{UFR}),$$

where RFR and RSC are the rinsed, liquid (decantate) flow rate and solute concentration and UFR and USC the unrinsed, decantate flow rate and solute concentration. When the effect of rinsing is merely to dilute the decantate, then $X = 0$; when the rinse does not dilute the decantate, $X = 1$, and the solute concentration is unaffected by rinsing. $X$ is probably slightly dependent on RFR; it would be greatest at the lowest RFR, 23 in this series. Using $\mathrm{RFR} = 23$ we estimate an upper

limit of 0.426 for $X$, and also using the highest RFR for this series, 51, gives $X$ an upper bound of 0.506. The median $X$ value, 0.46, was used to estimate the concentrations shown in Fig. 1.

Flow rates from the centrifuge for the second decanter run were calculated from the weight of collected flows for intervals. The rates are listed in Table 1 along with the compositions of samples taken from the collected centrifuge product streams. The feed rate, as deduced from measurements of the last two periods was 72 kg/h. The initial period indicates 13 kg/h of that is due to the solid collected in the decanter. The decanter holdup is approximately 5 kg. Liquid and solid samples were taken at the beginning and end of each interval and the composition average of those samples are listed for the interval. The liquid output slowed slightly during the middle two intervals of the run and the solids and lipid content of the liquid product increased during the run. The solid stream rates and solid content was steady, but the protein content decreased to zero during the middle interval, which is interesting but probably only indicates the difficulty of getting an accurate solid sample and analysis. The overall run average for the liquid stream was within 10–15% of the average for any interval.

The results of the third decanter run are listed in Table 2. Using the protein concentration values in the decantate, decantate flow rates for the rinsed and final unrinsed periods in the solute (protein) mass balance formula we calculated a displacement fraction of only 0.17 for this run. The higher feed flow rate produced a thicker extract layer in the decanter bowl that the rinse was not effective in displacing.

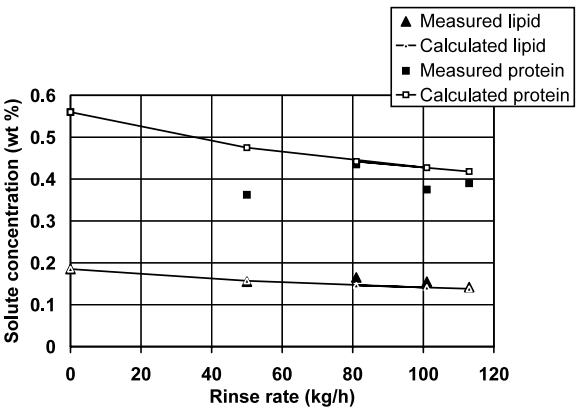


Fig. 1. Variation of centrifugate solute concentration with rinse rate for maize extract fed at 58 kg/h.

### 3.3. Settling from the extract into water

#### 3.3.1. Settling 226 kg of extract

Milled corn, 45 kg, was extracted with 181 kg of 55% ethanol and fed onto a plate resting at the top of 219 kg of water. The balls in the flow distributor did not have enough space to move and so the assembly operated as a fixed distributor plate. It was also apparent that sufficient water was not present at the bottom of the tank to allow the corn to settle completely below the upper surface of the water layer. The shortage of water became evident after 42 kg of ethanol had been pumped from the top of the deep end (after tipping tank to a 6° angle), and the liquid surface subsided below the corn on the shallow end. Thus, 90 kg of water was added to the tank at the bottom of the deep end.

As the liquid was pumped from the top of the settling tank, the composition and weight of sampled batches were determined. The measurements were used to calculate the values in Table 3. The recovered mass was 14.9 kg (3%) less than put into system: 492 kg of corn ethanol and water. Approximately 19 kg ($\sim$19%) of the ethanol was still associated with the solids or otherwise lost. About 46% of the ethanol was recovered at its original concentration and 57% was recovered at $\sim$37% in aggregate, close to the results of earlier runs. Nearly all of the solids and protein recovered from the liquid pumped off the top was in the first few batches. Mixing the extract and water layers reduces protein recovery. The lipid and starch concentrations were too low for useful interpretation.

#### 3.3.2. Settling 340 kg of extract

A portion, 159 kg (58% of the original extract), of the extract was recovered at nearly the original ethanol content, and 204 kg at 89% of the original. Increasing the scale of testing may improve ethanol recovery because of reduction of startup effects. It was evident that extract/water mixing occurs at the beginning when the extract layer (in

Table 1
Rates and composition of second decanter-centrifuged maize extract

| Sample[a] | Decanted liquid[b] (%) | | | | Decanted solids[b] (%) | | | Production rate (kg/h) | | Time, cumulative (min) |
|---|---|---|---|---|---|---|---|---|---|---|
| | Solids | Ethanol | Protein | Lipid | Solids | Protein | Lipid | Liquid | Solid | |
| 1 | 1.19 | 55.3 | 0.55 | 0.20 | 54.2 | 0 | 2.7 | – | – | 9 |
| 2 | 1.23 | 57.8 | 0.63 | 0.27 | 57.1 | 4.8 | 3.7 | 62.8 | 43.4 | 30 |
| 3 | 1.23 | 57.1 | 0.66 | 0.20 | 55.3 | 2.6 | 3.2 | 85.4 | 24.9 | 65 |
| 4 | 1.22 | 57.3 | 0.65 | 0.23 | 55.0 | 0 | 3.3 | 82.2 | 22.0 | 90 |
| 5 | 1.34 | 55.3 | 0.65 | 0.23 | 54.7 | 0 | 3.5 | 79.5 | 23.9 | 115 |
| 6 | 1.21 | 54.7 | 0.65 | 0.21 | 53.2 | 2.9 | 3.7 | 87.4 | 22.9 | 131 |
| 7 | 1.38 | 55.3 | 0.71 | 0.24 | 55.2 | 2.7 | 4.0 | 85.8 | 22.1 | 165 |

[a] The decanter was fed with 72 kg/h (extract mixture) and 35 kg/h (70% ethanol rinse).
[b] Each result is the average of two measurements.

the settling tank) forms. The extract liquid pumped off the settling tank the first day contained 1.6% solids and the overnight-settled extract had 1.1%. The average ethanol concentrations of cumulative liquid pumped from the top port of settling tank starting with the liquid pumped out while extract was being pumped into the lower port of the tank are shown in Fig. 2. As in the previous run, the settled extract liquid pumped out while extract liquid was being pumped in was undiluted.

### 3.3.3. *Settling 455 kg of extract*

The 164 kg of settled extract liquid pumped out of the upper port while extract was pumped to the tank had the same ethanol content as the unsettled extract liquid. The first 39 kg of the extract liquid that was recovered after settling overnight was diluted only slightly. The first 62% of the ethanol recovered from the settling tank was in a solution 90% of the original concentration. Because of greater extract mass, a greater undiluted extract liquid recovery compared with the preceding run was expected; however; recoveries of the two runs were equivalent.

## 4. Discussion

Recovery of solvent from the extracted corn can be accomplished by evaporation, and subsequent condensation for reuse. The heat of vaporization

Table 2
Rates and composition of third decanter-centrifuged maize extract

| Sample[a] | Decanted liquid[b] (%) | | | Decanted solids[b] (%) | | | Production rate (kg/h) | | Time, cumulative (min) |
|---|---|---|---|---|---|---|---|---|---|
| | Solids | Protein | Lipid | Solids | Protein | Lipid | | | |
| 1 | 1.50 | 0.65 | 0.21 | 57.9 | 4.26 | 3.30 | 94 | 36 | 7 |
| 2 | 1.72 | 0.71 | 0.25 | 55.8 | 3.61 | 2.21 | 94 | 36 | 17 |
| 3 | 1.55 | 0.69 | 0.24 | 56.1 | 4.28 | 1.94 | 94 | 36 | 27 |
| 4 | 1.29 | 0.57 | 0.23 | 56.3 | 4.11 | 2.64 | 136 | 41 | 33 |
| 5 | 1.30 | 0.55 | 0.25 | 55.8 | 3.43 | 2.66 | 136 | 41 | 42 |
| 6 | 1.30 | 0.56 | 0.25 | 55.9 | 3.01 | 2.60 | 136 | 41 | 56 |
| 7 | 1.73 | 0.72 | 0.30 | 56.1 | 3.92 | 1.52 | 98 | 43 | 67 |
| 8 | 1.73 | 0.73 | 0.36 | 56.3 | 5.47 | 2.21 | 98 | 43 | 77 |
| 9 | 1.74 | 0.74 | 0.31 | 56.5 | 3.08 | 2.33 | 98 | 43 | 83 |

[a] The decanter was fed with 2.2 l/min (extract mixture) for all samples and 35 kg/h (70% ethanol rinse) for the period covered by samples 4–6.
[b] Each result is the average of two measurements.

Table 3
Cumulative composition of extract liquid removed from settling tank

| Extract liquid removed (kg) | Component of extract liquid (%) | | | Component accumulated in extract liquid recovered (%) | | |
|---|---|---|---|---|---|---|
| | Ethanol | Solid | Protein | Ethanol | Solid | Protein |
| 41.8 | 48.2 | 1.75 | 0.70 | 20.1 | 30 | 47 |
| 70.9 | 45.2 | 1.50 | 0.57 | 32.1 | 44 | 64 |
| 98.8 | 42.5 | 1.29 | 0.44 | 42.0 | 53 | 70 |
| 127.3 | 39.8 | 1.13 | 0.36 | 50.7 | 60 | 74 |
| 154.4 | 36.9 | 1.02 | 0.31 | 57.0 | 66 | 78 |
| 180.5 | 36.3 | 1.00 | 0.31 | 65.5 | 75 | 89 |
| 208.9 | 33.4 | 0.91 | 0.27 | 69.9 | 80 | 92 |
| 236.7 | 31.1 | 0.85 | 0.25 | 73.6 | 84 | 94 |
| 261.5 | 29.0 | 0.80 | 0.23 | 76.0 | 88 | 96 |
| 286.3 | 27.6 | 0.78 | 0.21 | 79.0 | 93 | 98 |
| 310.5 | 26.1 | 0.77 | 0.20 | 81.0 | 100 | 100 |

Listed values include fines.

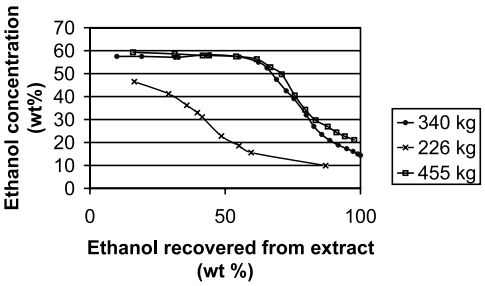


Fig. 2. Cumulative ethanol concentration of settled maize extract with recovery.

of 70% ethanol extract is about 1300 kJ/kg. Evaporating 1 kg of solvent from a 50% solids drained extracted maize will require about 1.1 kg of 1 MPa steam at a cost of approximately \$0.005. Zein comprises 5% of the maize mass and water 15% so that the, mostly starch, solid from which the ethanol solution is being recovered will be about $80/5 = 16 \times$ the mass of the extracted zein. Therefore, the steam cost to evaporate solvent from the extracted and drained (to 50% solids) maize will be at least \$0.08/kg of zein. When the draining is less efficient, for example using a screen rather than a decanter, then the evaporation energy cost to recover the ethanol will be greater.

In addition to the cost of evaporation, solute in the liquid will remain with the corn and thus the dissolved zein will be lost a result of evaporation. With a 4:1 ratio of extracting liquid to corn and a screened or unrinsed decanted liquid content of 50%, one-fourth of the zein extracted will be left on the extracted corn after evaporation.

### 4.1. Packed bed rinsing

We attribute the low permeability of the extract bed to the size reduction of the milled corn by the extraction process (30–50 μm) and possibly also to their increased plasticity that produces a bed with fine tortuous pores. Displacement methods involving thick layers of extracted corn appear to be too slow to be practical even though relatively low in capital cost.

### 4.2. Decanter centrifuge rinsing

Rinsing during decanter centrifugation is not effective for fine particles because the cake formed is fairly impermeable to the rinse (Dahlstrom, 1997). Thus, the rinsing while centrifuging method suffers from the same problem as the packed bed displacement method. Decanter centrifuge rinsing was nearly 50% effective at a rinsing rate about the same as the feed flow rate (58 kg/h). At the higher feed flow rates of the second and third trials with rinse rates of 35 kg/h, the protein recovery in the liquid stream decreased to 46% at 72 kg/h and 43% at 140 kg/h, which is about the same as the

unrinsed recovery. These reduced rinsing effects are consistent with the decrease in displacement with rinse/feed rate ratio shown by the results of the first run (Fig. 1).

Even at the lowest rate, the solid product would need to be rinsed with water at the end to recover the ethanol solution used in the first rinse. To prepare a pumpable mixture (with a solid content of 30% or less) for water rinsing, the ethanol-rinsed, decanted stream containing the solids would have to be mixed with additional 70% ethanol.

### 4.3. Settling to water

The primary function of this method is to transfer most of the extracted corn particles to the water with minimal mixing of the extract solution and water. To achieve this, the extract mixture must be pumped carefully onto the water, but at a practical rate, to dissipate most of the extract fluid momentum in the extract liquid layer. In a steady process, the extract will have to be pumped from the layer at the same rate as it is pumped to it with sufficient holdup to permit settling of the particles to the water layer. From an equipment cost standpoint, however, it is desirable to keep the liquid layers thin so that the time for particles to descend through them will be short.

For a continuous steady process, water containing the settled solid particles must be pumped out of the settling tank to prevent solid particle accumulation on the bottom of the tank and interference with the transfer of the particles to the water. Particles can be removed from the water outlet stream with screens or hydrocyclones, when necessary, and then pumped to downstream liquefaction. The water flow rate must be large enough to keep the settled particles from accumulating at the tank bottom, but small enough to avoid mixing the liquid layers. In these settling experiments, the settled extract liquid contained 1–2% solids, whereas the extraction mixture was approximately 20% solids, indicating that the conditions used were effective, but not necessarily optimal.

The settling method has some advantages unrelated to separation efficiency. It is relatively insensitive to the temperature and solvent content of the feed. The decanter centrifuge, for example, requires ethanol-compatible journal bearings for continuous admission of ethanol extracts. Because of the gradual way the particles are separated from the liquid, the particles could, in principle, be classified by collecting them with more complicated settling equipment permitting removal of particles at different positions in the water layer (Masliyah et al., 1981). The settling method will produce a liquid extract with more fine solids than the decanter. This may be desirable for zein recovery when a substantial quantity of protein bodies is left in the liquid.

## 5. Conclusions

Displacement of the extract liquid from a packed bed of extracted corn is limited by low permeation rates. For centrifuge rinsing, about one-half of the extract liquid (as measured by protein) that would be lost without rinsing, could be recovered when the rinse feed was about the same as the extract feed rate. When rinsing rates was one-fourth of the extract feed rate, the protein recovery was 44%, with or without rinsing, and 80% of the extract liquid was recovered undiluted. An undiluted recovery of 58% was obtained by controlled settling of the extract into a water layer. An additional 23% of the extract liquid was mixed with water forming a layer between the extract and water layers.

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